# Initial Study of Novel Flame Retardants For Epoxy Resin Systems

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## Abstract

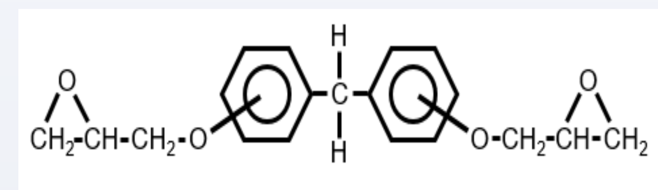
This poster describes the initial investigation of the reaction between a novel flame retardant (FR) and epoxy resin. The novel chemical (synthesized and provided by UD Chemistry Department faculty) is a phosphorous based flame retardant that also incorporates primary and/or secondary amine functional groups. These groups potentially could react with epoxy resins, which would then incorporate this FR chemical directly into the polymer network through covalent bonding. This would be a way of introducing flame retardants into epoxy resins to improve flammability of composites. Initial research was conducted using Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR). The results indicate that the flame retardant is indeed reacting with the epoxy resin to form a crosslinked network. Future work will involve characterization of the cured epoxy-FR network for physical properties, mechanical properties, and flammability

## Introduction

Epoxy resin systems are one of the most common thermosetting polymers used in several industrial fields as adhesives, coatings, and composite matrices. They have been used as high-performance materials because of excellent mechanical properties and chemical resistance, good adhesive strength, low cure shrinkage, and can be formulated with appropriate rheology for surface coatings. However, the flammability of epoxy resins has critically limited their uses in many applications such as electronics, aerospace, and transport vehicles. In the recent years, several type of flame retardants have been developed for epoxy resins, although some of these are in the form of separate additives that can phase-separate or leach out over time. Phosphorus compounds are able to form a char layer on the surface when a threshold temperature is reached in a fire situation. The char layer imparts fire resistance by forming a thermal and diffusion barrier at the surface. In this study, the reaction of the flame retardant (P_Et_H) with an epoxy resin was investigated by Differential Scanning Calorimetry (DSC), and Thermogravimetric Analysis (TGA).

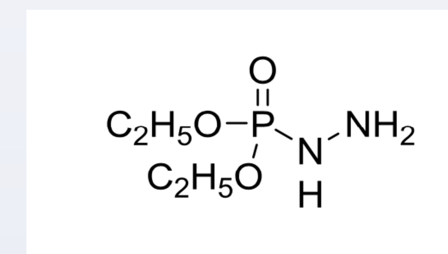
## Materials

I. Epoxy



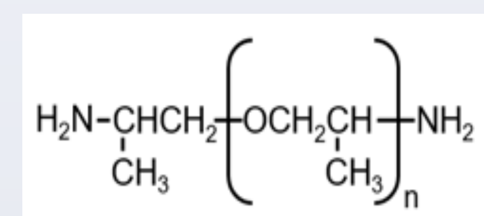
**EPON 862 (liquid)**
EEW[1] = 169 g/eq

II. Phosphoryl hydrazide flame retardant (FR)
(synthesized by UD Chemistry Dept. faculty)



**P_Et_H (liquid)**
P = 18.4 wt%
AHEW[2]=56 g/eq

III. Traditional Curing Agent



**Epikure 3274 (liquid)**
(n=5.6)
AHEW[2] = 76 g/eq

Mixture #1 (I + II)

Mixture #2 (I + II + III)

[1]EEW = epoxide equivalent weight [2]AHEW = amine hydrogen equivalent weight

## Experimental Procedure

**Mixture #1:**

- This was prepared to investigate the basic cure reaction between the FR and epoxy.
- It was comprised of a stoichiometric balance between epoxide and N-H groups (75 wt% EPON, 25 wt% P_Et_H)

**Mixture #2**

- This was prepared to evaluate how the FR and traditional curing agent could co-cure with epoxy.
- It was formulated to have a stoichiometric balance between epoxide and total N-H groups while maintaining the “active ingredient” (P) at a level of 2.5 wt%. (72.3 wt% EPON, 13.6 wt% Epikure, 14.1wt% P_Et_H)

## Results and Discussions

- DSC and TGA results for freshly mixed samples are given below. These illustrate the cure reaction between epoxy and the flame retardant, as well as the flame retardant reaction (decomposition of P_Et_H to form char)
- Additional samples (not shown here) were aged for various times and temperatures up to 120°C. These hardened over time, indicated that cure had taken place.

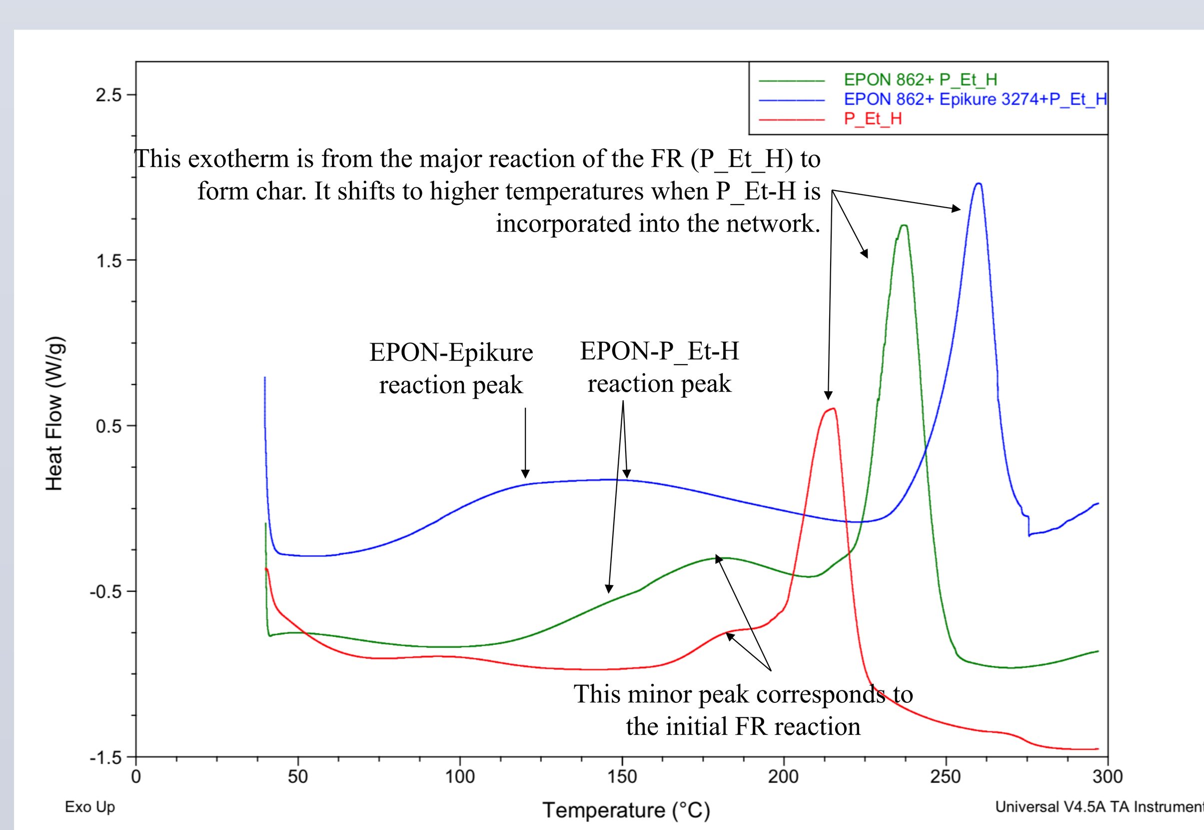



Fig. 1. DSC results for epoxy-flame retardant mixtures.

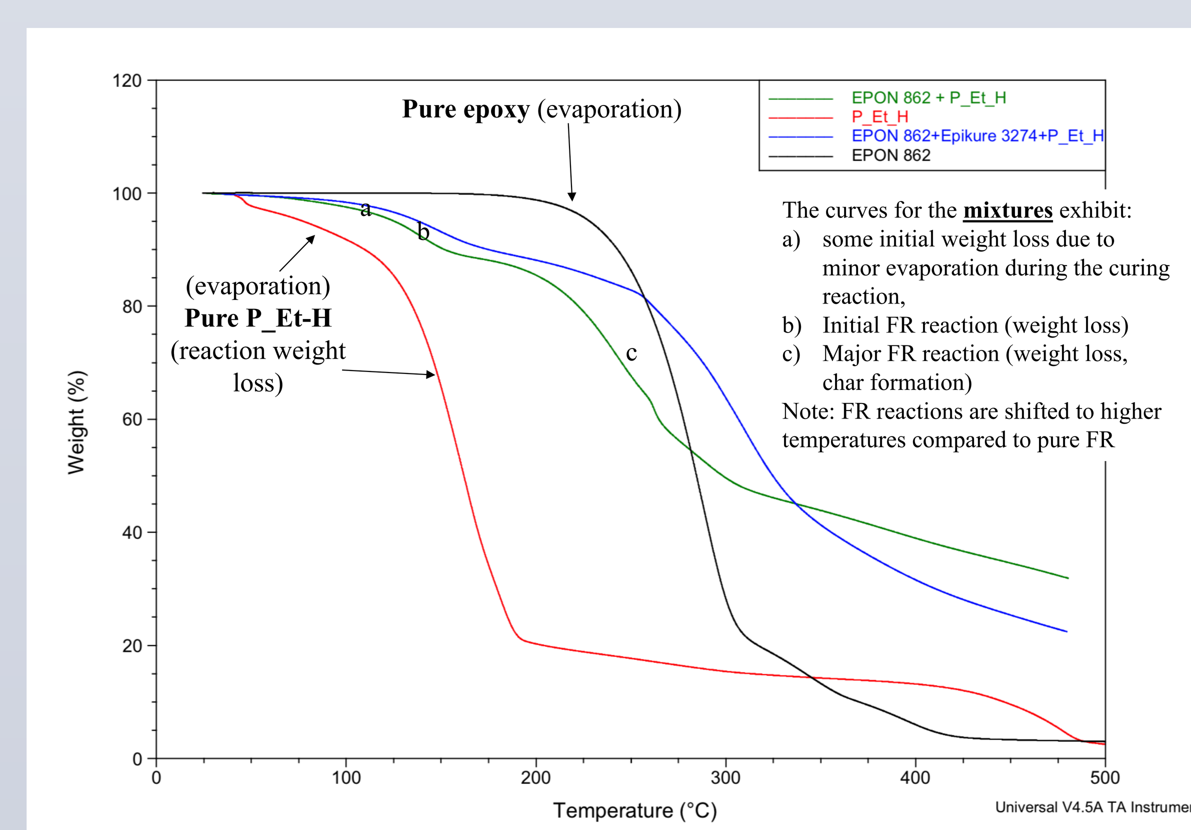



Fig. 2. TGA results for epoxy-flame retardant mixtures.

## Conclusion

Both DSC and TGA results indicate that the flame retardant (P_Et_H) cured with the epoxy resin to form a thermoset polymer through the reaction between epoxy and amine groups of P_Et_H. This reaction also occurred when Epikure 3274 was used as a co-curing agent. Both DSC and TGA results also indicated that the flame retardant reaction (decomposition of P_Et-H) was shifted to higher temperature as a result of being incorporated into the network. The addition of a co-curing agent (Epikure 3274) resulted in an increase of decomposition temperature of the epoxy resin-flame retardant and appears to have affected the epoxy cure chemistry More study is needed prior to scale-up of the material for flammability testing.

## Future Work

I. Flammability of cured epoxy resins.
II. Morphology of the residue.
III. Mechanical properties
IV. Rheology of cured epoxy resins.

## Faculty Advisors / Collaborators

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